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TECHNICAL REPORT

EVALUATION OF ELASTOMERS FOR
POTENTIAL USE AT CRYOGENIC TEMPERATURES

By

19960426 016

J. D. Ruby

Department of the Army Project No. 1-H-0-24401-A-110

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Date 13 May 1963

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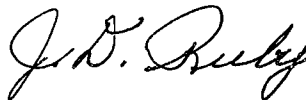
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Approved by:



A. C. HANSON
Laboratory Director

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ABSTRACT

The purpose of this work was to determine the lowest temperature at which a variety of rubber vulcanizates and other nonmetallic materials will remain nonbrittle and flexible and exhibit moderate recovery from applied deformation.

The operation of a mechanically refrigerated low temperature test cabinet useful at temperatures down to -320°F is described.

RECOMMENDATIONS

It is recommended that further work be conducted to improve the low temperature brittleness and flexibility of rubber at temperatures of -100°F and below.

**EVALUATION OF ELASTOMERS FOR
POTENTIAL USE AT CRYOGENIC TEMPERATURES**

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EVALUATION OF ELASTOMERS FOR POTENTIAL USE AT CRYOGENIC TEMPERATURES

OBJECT

To determine, by conventional ASTM test procedures, the lowest temperatures at which a variety of rubber vulcanizates and other nonmetallic materials will remain nonbrittle and flexible and will exhibit moderate recovery from applied deformations.

To ascertain if the ASTM test method and equipment currently being used to determine the brittleness, flexibility and elastic recovery of rubber down to -100°F are applicable at temperatures ranging from -100 to -300°F .

To determine the variations in temperature between various points and the fluctuations at any given point within the chamber of a mechanically refrigerated low temperature test cabinet.

INTRODUCTION

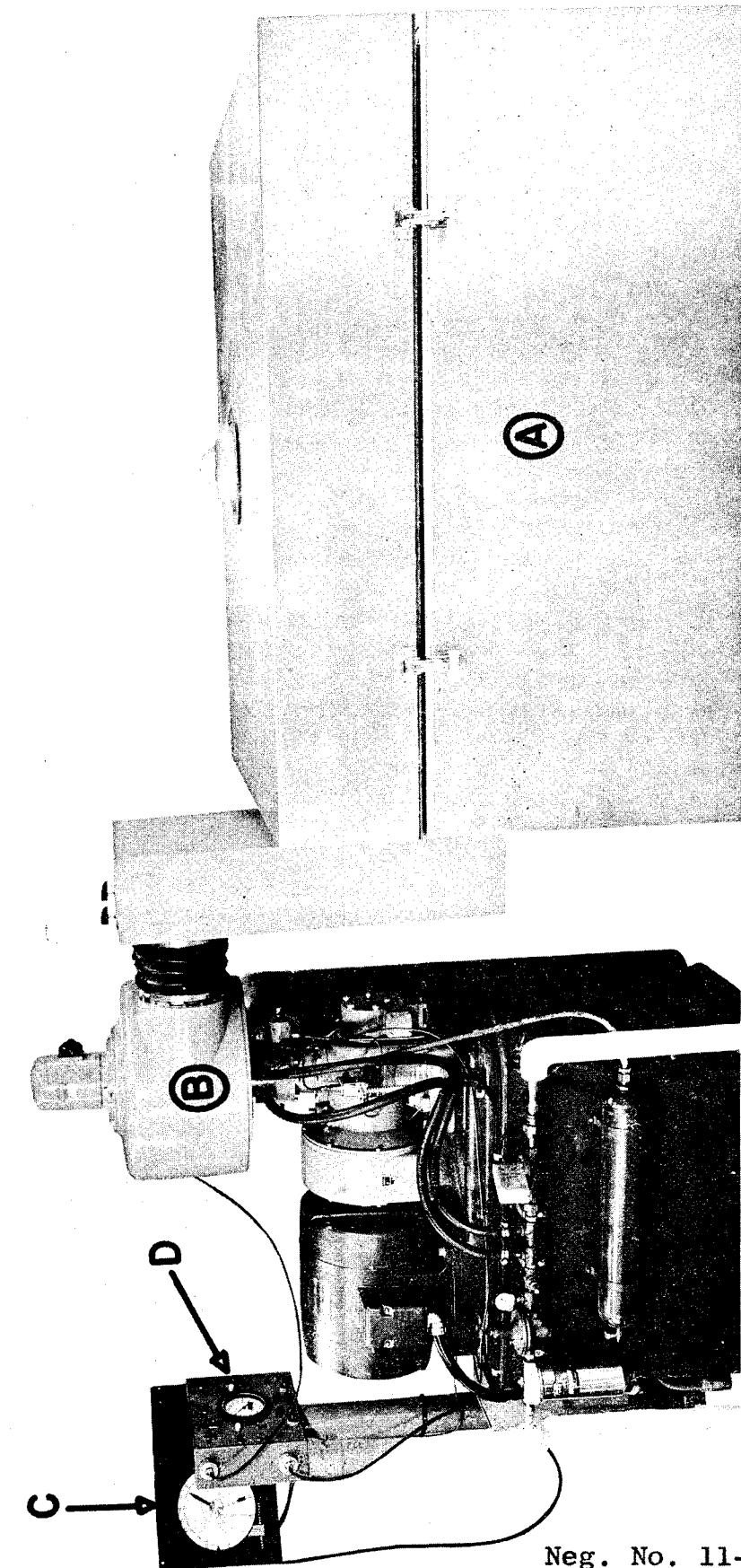
A great deal of effort has been expended by the Military toward the development of rubber items suitable for use at temperatures down to -65°F and capable of being stored at temperatures as low as -80°F . Only very rarely have uses or storage requirements at temperatures below these been established. Consequently, little information is presently available on the properties of elastomers at -100°F or below.

In the face of rapidly advancing missile technology, it is important that the cryogenic potential of available elastomers be exploited to the utmost. The need for elastomers serviceable at temperatures of -100°F or below arises from two principle potential applications: (1) elastomeric sealants in contact with cryogenic fuels or oxidizers and (2) elastomers for use at temperatures of outer space.

PROCEDURE

An environmental system consisting of a specially insulated exposure chamber and a cryogenerator were used for the tests described in this report. (Figure 1)

The exposure chamber has a working space of 2 x 2 x 3 feet. It is surrounded by 12 inch thick insulated walls.



OVERALL VIEW OF CRYOGENERATOR

- A - Exposure chamber
- B - Heat exchanger
- C - Controller recorder
- D - Control panel and helium pressure gauge

FIGURE 1

The lid has a six inch access port through which test equipment may be operated.

The times required to cool the exposure chamber to various temperatures were determined using a thermocouple and a portable potentiometer.

To determine the lowest temperature which could be obtained, the temperature controller (C - Figure 1) was disconnected, thus allowing the cryogenerator to operate continuously at maximum capacity. Five hours after starting, a temperature of -321°F was reached, and continued operation for an additional hour produced no further temperature reduction. A curve of temperature vs operating time is given in Figure 2.

To determine temperature variations between different locations within the chamber, the cryogenerator was allowed to cycle two or three times and temperatures were determined at each position shown in Figure 3. Maximum and minimum temperatures recorded at these positions, while the box was operating at various temperatures, are shown below the diagram.

In order to determine if exposure of the ASTM D746 solenoid brittleness tester to temperatures ranging from ambient to -300°F would have a serious effect on the speed of the striker, speed checks were made employing the modified impacted ball method for determining speed.⁽¹⁾ These results are given in Table I and indicate that when the entire apparatus is subjected to low temperature, a reduction in striker speed results. The reduction in speed was evident at -40°F and became progressively larger as the temperature was lowered. At -300°F , a total reduction in speed of 18 percent had occurred. From previous work on impact speed vs brittle temperature,⁽²⁾ it was estimated that this speed reduction could cause an 8°F shift in brittle point temperature. The possible 8°F error in brittle point temperature at -300°F was considered acceptable as this is less than the variation in temperature within the chamber at -300°F . The reduction in striker speed and subsequent error in brittle point was probably due to an increase in friction resulting from uneven contraction in the metal components of the apparatus.

Flexibility of elastomers at extreme low temperature was determined by test method ASTM D1043. This method was selected over ASTM D1053 procedure because it was more readily adaptable for use within the cryogenerator. The method worked very well at temperatures down to -300°F . Due to stiffening at these low temperatures, the nylon cords which hold the weights had to be replaced with linen.

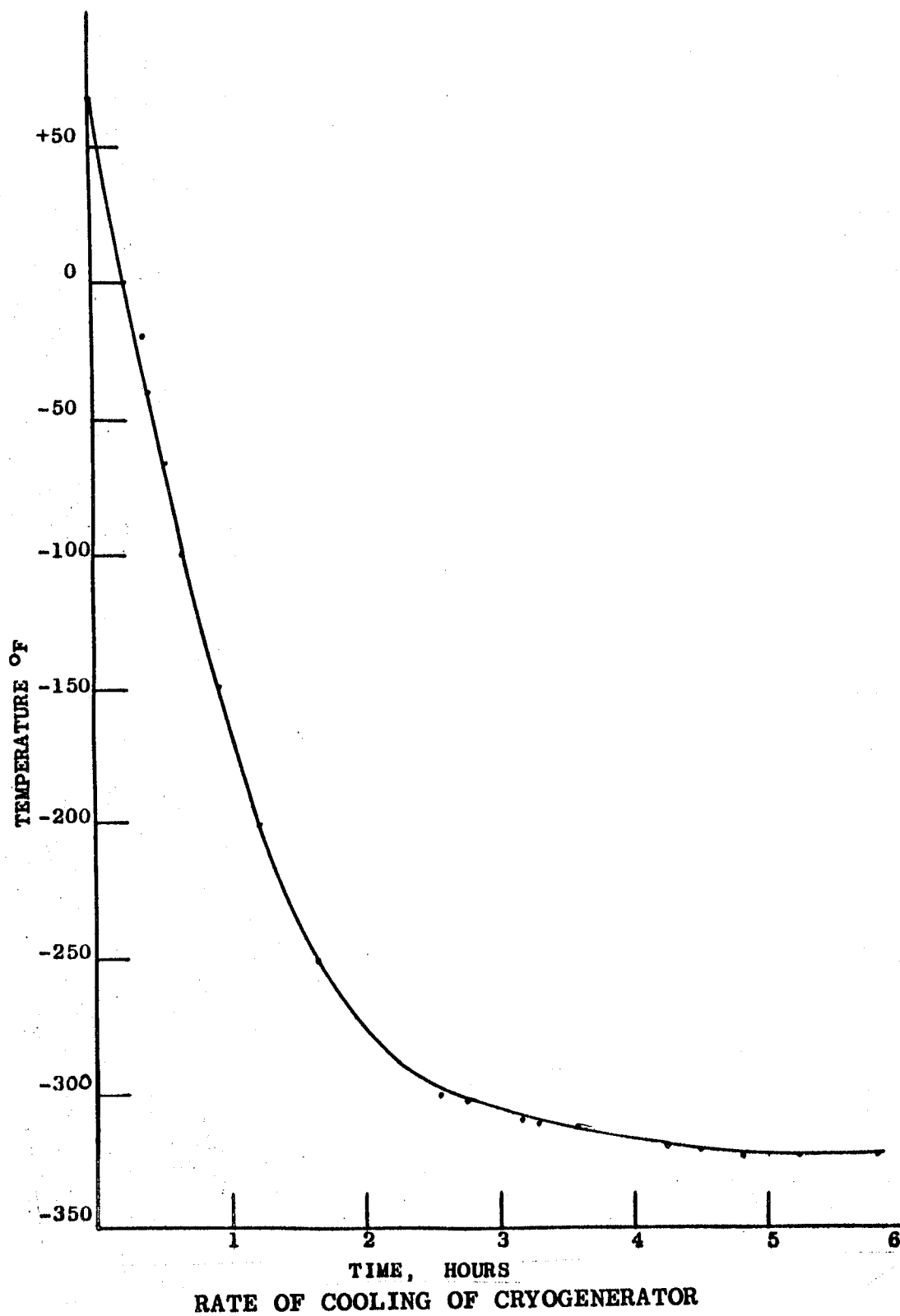
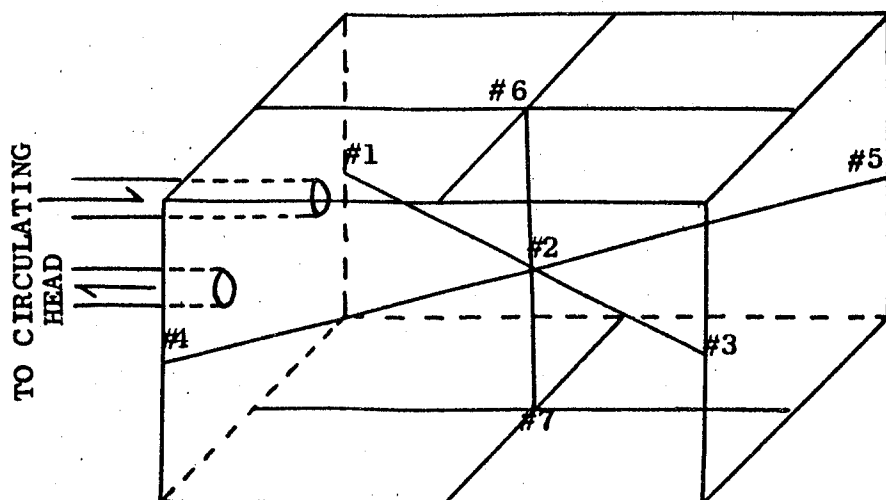


FIGURE 2



POSITIONS AT WHICH TEMPERATURE WAS RECORDED

RECORDER TEMPERATURE	POSITION 1	POSITION 2	POSITION 3	POSITION 4	POSITION 5	POSITION 6	POSITION 7
0°F	+4 -0	+2 -3	+2 -4	+3 -2	+4 -3	+2 -3	+2 -4
-20°F	+2 -4	+0 -4	+0 -5	+1 -5	+2 -4	+3 -4	+1 -5
-40°F	+2 -4	+4 -3	+4 -4	+4 -4	+5 -2	+4 -4	+4 -3
-67°F	+3 -3	+3 -4	+3 -4	+1 -5	+1 -6	+1 -7	+0 -8
-100°F	+2 -4	+3 -4	+2 -5	+4 -4	+4 -4	+4 -4	+5 -4
-150°F	+2 -4	+2 -6	+2 -6	+2 -6	+2 -7	+3 -6	+2 -6
-200°F	+4 -4	+4 -5	+3 -7	+4 -6	+2 -8	+5 -6	+6 -7
-250°F	+6 -4	+6 -4	+6 -5	+6 -5	+6 -4	+5 -5	+7 -4
-300°F	+6 -2	+7 -2	+8 -3	+7 -2	+7 -2	+8 -3	+8 -3

TEMPERATURE VARIATION WITHIN CHAMBER

TABLE I

SOLENOID SPEED AT VARIOUS TEMPERATURES

<u>TEST</u> <u>TEMPERATURE</u>	<u>TRIAL</u> <u>1</u> <u>FT/SEC</u>	<u>TRIAL</u> <u>2</u> <u>FT/SEC</u>	<u>TRIAL</u> <u>3</u> <u>FT/SEC</u>	<u>TRIAL</u> <u>4</u> <u>FT/SEC</u>	<u>TRIAL</u> <u>5</u> <u>FT/SEC</u>	<u>AVERAGE</u> <u>FT/SEC</u>
Control Room Temp.	10.1	9.7	9.5	10.1	9.7	9.8
Chamber Room Temp.	9.8	9.8	9.9	10.0	9.7	9.8
Chamber -40°F.	9.9	9.5	10.1	9.8	8.7	9.6
Chamber -67°F.	8.7	8.2	10.2	9.1	9.5	9.3
Chamber -100°F.	8.9	8.7	8.4	9.3	8.5	8.8
Chamber -150°F.	8.3	9.2	8.5	8.6	8.9	8.7
Chamber -200°F.	8.9	8.0	8.3	9.7	8.8	8.7
Chamber -250°F.	9.5	9.2	8.0	8.2	9.5	8.9
Chamber -300°F.	7.8	8.3	8.2	8.1	7.6	8.0

Elastic recovery of elastomers at low temperatures was determined by ASTM Method D1229 for low temperature compression set instead of the temperature retraction method because the exposure chamber was not equipped with heaters for controlled warm-up.

The low temperature properties of various unplasticized elastomers are presented in Table II. This table served as a guide for selection of polymers which, through proper compounding and/or chemical modification, might be serviceable at temperatures of -100°F or below.

Ten polymers exhibiting the most promising potential for use at low temperatures were selected for further testing.

All compounds except the castable polyurethane were mill mixed on an 8 x 18 inch, two roll mill, using formulations given in Table III. The castable polyurethane was prepared by the following procedure:

1. Add polyurethane liquid to a resin flask and heat to 212°F with stirring.
2. Degas liquid at 212°F and a maximum pressure of 5 mm. Hg.
3. Add 4,4'-methylene di-o-tolyl(2 chloro aniline) as a liquid at 212°F .
4. Mix under vacuum for 3 minutes at 212°F .
5. Pour into mold and cure 3 hours at 212°F , then post cure 14 days @ 75°F and 50% R.H.

Specimen preparation and tests were in accordance with ASTM procedures where applicable.

Time required for a specimen to reach equilibrium after being exposed to low temperature was determined (Figure 4). It was found that a minimum of 10 minutes was required for the specimen to reach the temperature of the exposure chamber and this time was used on all subsequent brittleness and flexibility determinations.

Low temperature brittleness and flexibility were determined on two types of leather and one type of paper, since these materials were believed to be flexible at extremely low temperatures.

It was found that both materials remained nonbrittle at -300°F and were sufficiently flexible to be easily bent and

TABLE II

**LOW TEMPERATURE BRITTLINESS AND FLEXIBILITY
DATA FOR VARIOUS TYPES OF ELASTOMERS**

<u>ELASTOMER TYPE</u>	<u>ASTM D1043 T₂₀₀ °F</u>	<u>ASTM D746 °F</u>
Methyl phenyl silicone *	-110	Below -130
Cis-1,4-polybutadiene *	Below -90	
Methyl phenyl vinyl silicone	Below -80	Below -80
Butadiene-styrene 76.5/23.5	-80	Below -65
Fluorosilicone	-78	Below -90
Butadiene-styrene 92/8 *	Below -77	Below -67
Synthetic natural	-66	Below -60
Ethylene propylene *	-64	Below -90
Natural rubber	-62	-70
Nitroso rubber	-47	-
Polyurethane (polyether-millable)	-46	Below -80
Carboxy modified butadiene	-42	Below -72
Polyurethane (polyester-millable)	-40	Below -80
Butadiene acrylonitrile 82/18	-34	-50
Polychloroprene	-21	-48
Polysulfide	-20	Failed -65
Polyurethane (polyether-castable)	-16	Below -85
Butadiene acrylonitrile 74/26	-16	-36
Butadiene acrylonitrile 65/35	-2	-12
Butadiene acrylonitrile 55/45	+29	+18

* Literature Value

TABLE III

COMPOUNDING FORMULATIONS

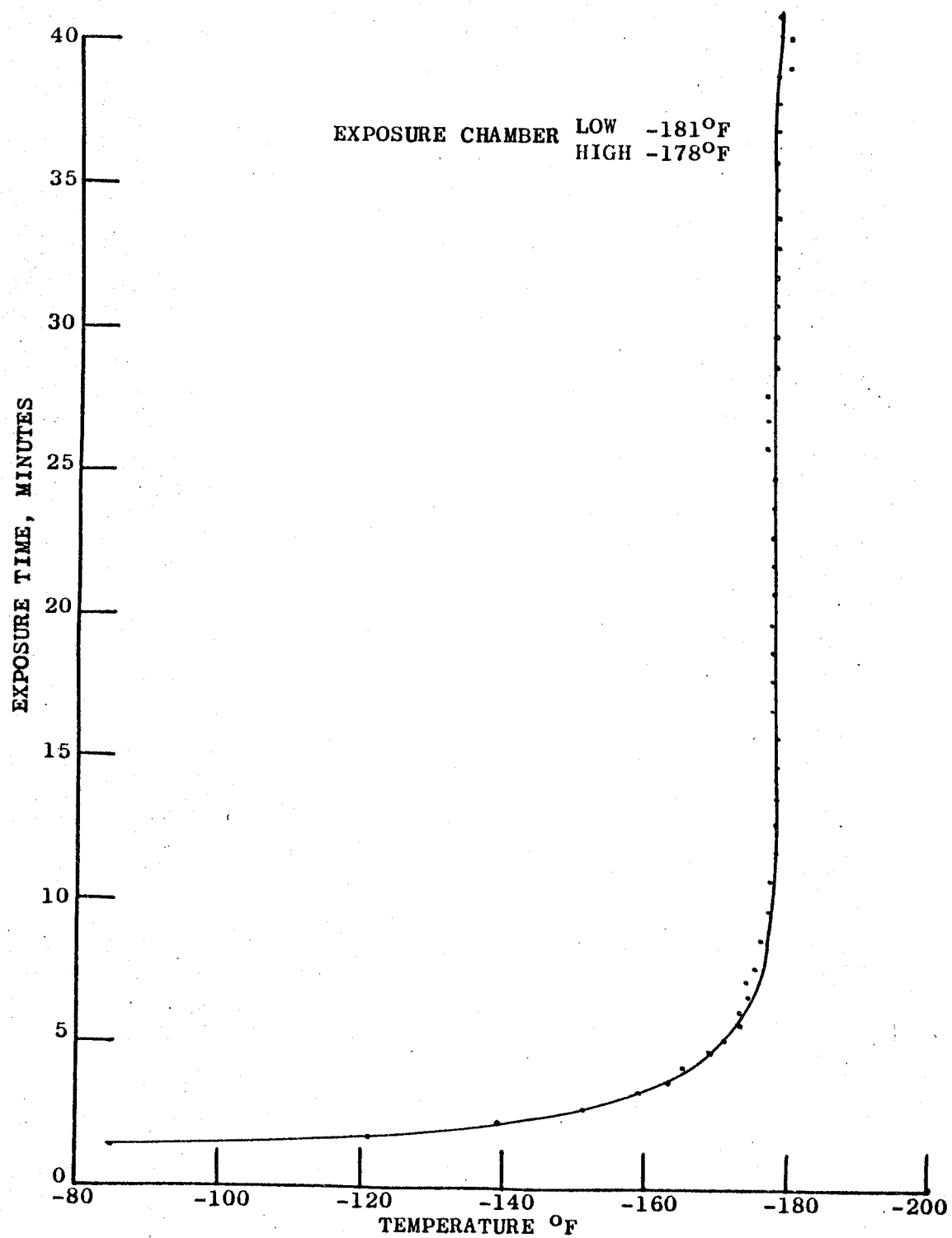
COMPOUNDING INGREDIENT	Z56B	Z54	Z81F1	A25	B1	Z103 CIF3B	Z107	S103	Z46E	Z55P
Methyl phenyl vinyl siloxane	100									
Methyl phenyl siloxane		100								
Fluoro silicone			100							
Synthetic natural										
Cis 1,4-polybutadiene				100	114	100	100	100	100	100
Carboxy modified butadiene										
Ethylene propylene										
Butadiene-styrene 92/8										
Polyurethane(polyether) *				3	5	5	5	5		
Polyurethane(polyether) **				2	2	2		1.5		
Zinc oxide							1			
Stearic Acid										
Calcium stearate										
1,2-dihydro 2,2,4-trimethyl quinoline				1		3				
Phenyl-beta-naphthylamine				1	1					
Petroleum wax mixture						2				
Sulfur				1.75	1.75	1.75	0.32	2		
N-cyclohexyl 2-benzo thiazole sulfenamide				1	1	1				
Benzothiazyl disulfide							7	3		
Dicumyl peroxide on calcium carbonate silicone fluid	1.3		1.3						5	
Dichloro benzoyl peroxide in silicone fluid										
4,4' methylene di-o-tolyl isocyanate										
4,4' methylene bis 2 chloroaniline				50	50	30				11
FEF Black										
ISAF Black										
EPC Black										
Silica				8			60	30		
TOTAL PARTS	101.3	100	109.3	159.75	174.75	144.75	173.32	141.5	105	111

All Compounds were cured 30 minutes @ 307°F except:

Z54 Cure 10 min. @ 300°F Post Cure 24 hrs. @ 480°F
 Z81F Cure 10 min. @ 300°F Post Cure 24 hrs. @ 400°F
 Z56C3 Cure 10 min. @ 275°F Post Cure 8 hrs. @ 375°F
 Z46E Cure 20 min. @ 275°F Post Cure 4 hrs. @ 215°F
 Z55P Cure 3 hrs. @ 212°F Post Cure 14 days @ 75°F & 50% R.H.

* Millable

** Castable



SPECIMEN TEMPERATURE VS TIME OF EXPOSURE

flexed by hand. The temperature at which Young's Modulus was 10,000 psi was found to be -67°F and -130°F for the leather specimens, as determined by ASTM D1043 procedure. Both specimens of leather were chrome tanned cowhid, but were of different grades. One was a strap and harness grade, the other a Cordovan used in making shoe uppers. The difference in flexibility possibly comes from different finish treatment, such as type and amount of oil or filler used in the leather.

RESULTS AND DISCUSSION

Test results for the ten compounds selected are given in Table IV from which several conclusions can be drawn.

1. Eight of the ten compounds tested had brittle fracture temperatures of -100°F or below, but only three of ten remained sufficiently flexible to be considered serviceable at these temperatures.

2. Correlation between brittleness and flexibility was fairly good for six of the compounds, having an average difference of 140°F . For the remaining four compounds, the average difference was 72°F . The poor flexibility temperature as compared with the low brittleness temperature for the polyurethane compounds may be due to their high strength bath at room and low temperature.

3. The temperatures at which elastic recovery tests were performed were selected more or less at random and were not optimum temperatures for producing best results. The silicone, cis 1,4-polybutadiene and carboxy modified butadiene compounds have exceptionally poor recovery at temperatures well above those at which they became stiff or brittle. This was probably due to the development of crystallization during the longer exposure period.

CONCLUSIONS

This work reveals that even those elastomers having the best properties at low temperatures are limited to a range of approximately -185°F . The transition from this range to that of liquid nitrogen temperature, using ordinary compounding techniques, appears to be a remote possibility. Modification of those natural materials, such as leather or cellulose (which possess flexibility at liquid nitrogen temperatures) to incorporate a degree of elasticity, would appear to be one of the best approaches to obtaining a truly cryogenic elastomer.

TABLE IV
PROPERTIES OF RUBBER VULCANIZATES AND NONMETALLIC MATERIALS

PROPERTY MEASURED	METHYL PHENYL SILOXANE	METHYL PHENYL VINYL SILOXANE	FLUORO- SILOXANE	SYNTHETIC NATURAL	BUTADIENE 92 8	ETHYLENE PROPYLENE	CIS 1,4 POLY BUTADIENE	POLYURETHANE (MILLABLE)	POLYURETHANE (CASTABLE)	CARBOXY MODIFIED BUTADIENE	STRAP & HARNESS LEATHER	CORDOVAN LEATHER	KRAFT PAPER
Tensile Strength, psi.	Z54	Z56C3	Z81F1	A25	S103	Z107	B-1	Z46E	Z55P	Z103 C1F3B			
Modulus 300% E., psi.	530	1390	770	1410	2440	2400	1840	3740	5470	2480			
Elongation, %	200	380	610	810	360	1140	960	1690	2010	290			
Hardness Shore A-2	48	50	50	56	52	61	54	68	94	71			
ASTM D746 OF	-182	-179	-89	-70	-116	-100	-138	-100	-162	-110	OK	-300	OK-300
ASTM D1043, T200 OF	-176	-168	-82	-64	-88	-45	-110	-39	-22	-76	-67	-130	
ASTM D1229 Recovery After 30 Min., %	5	5	30	32	24	18	2	24	29	0			
Temperature at which D1229 was determined OF	-150	-150	-67	-67	-67	-40	-67	-40	-40	-40			

LITERATURE REFERENCES

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"Effect of Impact Speed on the Brittle Temperature of
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